ADA

EFFECTIVENESS OF LOW TEMPERATURE ADDITIVES FOR BIODIESEL BLENDS

INTERIM REPORT
TFLRF No. 428

by
Steven R. Westbrook

U.S. Army TARDEC Fuels and Lubricants Research Facility
Southwest Research Institute® (SwRI®)
San Antonio, TX

for
U.S. Army TARDEC
Force Projection Technologies
Warren, Michigan

Contract No. W56HZV-09-C-0100 (WD13)

June 2012

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14. ABSTRACT

The basis of this focus was the assumption that once the end user has taken ownership of a biodiesel blend, the only realistic option to improve low temperature properties is treatment of the blend with an additive. The test results demonstrated that use of the selected additives could be tailored depending on the low temperature property to be improved. Addition of additive to cold fuel reduces or eliminates the efficacy of the additive.

15. SUBJECT TERMS

Biodiesel, biodiesel blends, low-temperature operability, cold flow improver additives

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EXECUTIVE SUMMARY

The objective of this effort was to determine the effectiveness of available cold flow improving additives when added to various concentrations of biodiesel blends under a variety of conditions. The work reported herein was focused solely on the use of additives to improve low-temperature handling properties of biodiesel blends. The basis of this focus was the assumption that once the end user has taken ownership of a biodiesel blend, the only realistic option to improve low-temperature properties is treatment of the blend with an additive. The test results demonstrated that use of the selected additives could be tailored depending on the low-temperature property to be improved. Addition of additive to cold fuel reduces or eliminates the efficacy of the additive. The additives tended to perform equally in any biodiesel blend concentration although the magnitude of performance depended on biodiesel concentration. While there were some differences, the fatty acid profile did not have a large effect on additive efficacy.

FOREWORD/ACKNOWLEDGMENTS

The U.S. Army TARDEC Fuel and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, performed this work during the period June 2010 through June 2012 under Contract No. W56HZV-09-C-0100. The U.S. Army Tank Automotive RD&E Center, Force Projection Technologies, Warren, Michigan administered the project. Mr. Eric Sattler and Mr. Allen Comfort served as the TARDEC contracting officer's technical representatives. Luis Villahermosa and Lori Zuziak of TARDEC served as project technical monitor.

The authors would like to acknowledge the contribution of the TFLRF technical support staff along with the administrative and report-processing support provided by Dianna Barrera.

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ACRONYMS AND ABBREVIATIONS

B100............100% biodiesel

CFPP...........Cold Filter Plugging Point, ASTM D6371

CP................Cloud Point, ASTM D5773

CSFT...........Cold Soak Filterability Test, ASTM D6751

PP................Pour Point, ASTM D5949

USLD..........Ultra-Low Sulfur Diesel

1.0 BACKGROUND

The objective of this effort is to determine the effectiveness of available cold flow improving additives when added to various concentrations of biodiesel blends under a variety of conditions.

The U.S. Army is increasing its exposure to fuels containing biodiesels per the expanded use of B20 and the allowance of up to 5% biodiesel for fuels delivered under ASTM D975. As the availability of fuels containing biodiesel increases, so does the exposure of tactical equipment to these fuels and the potential problems associated with their use. The U.S. Army tactical equipment sees an operational profile that differs from the commercial fleets in a number of key areas. Army equipment has sporadic utilization and is subjected to long-term storage and prepositioning that can extend beyond 32 months. Furthermore, mileage accumulation by active equipment does not exceed 3,000 miles per year on average, and thus creates the potential for using fuel across different seasons. Very little work has been done to determine the efficiency and effectiveness of additives to affect low temperature properties with fuels containing biodiesel. Traditional additives were developed around suppressing wax formation and agglomeration, however, biodiesel is not a waxy or paraffinic compound, so the effectiveness of these additives needs to be understood. Also, new additives have been developed for biodiesel containing fuels but there is little information available on their effectiveness. Additional work is needed to understand these issues.

2.0 APPROACH

Under this project, biodiesels made from various sources, all of which conformed to ASTM D6751, were acquired. Three concentrations of each biodiesel were blended as follows: 5%, 10% and 20%. Four additives, designed to improve low temperature properties of biodiesel blends, were obtained and their effectiveness and efficiency determined. Test methods included Cloud Point (CP), Pour Point (PP), and Cold Filter Plugging Point (CFPP).

3.0 EXPERIMENTAL

3.1 Test Methods

The cloud point (CP, ASTM D5773) and pour point (PP, ASTM D5949) data were obtained using a Model PSA-70X analyzer manufactured by Phase Technology. The cold filter plugging point (CFPP, ASTM D6371) data was determined using a KLA-4-TS automatic tester. Cold Soak Filterability Tests (CSFT) were run according to the procedure in ASTM D6751-09, Annex A1.

3.2 Test Fuels

An attempt was made to obtain B100s that represented a variety of compositions. Numerous suppliers were contacted and the six (6) B100s listed below were eventually obtained for use in this work:

- CL10-1502 – canola-based
- CL10-1564 – soy-based
- CL10-1894 – mixed feedstock
- CL10-1897 – palm-based
- CL10-1898 – mixed feedstock
- CL10-1899 – mixed feedstock

Each of the B100s was analyzed for fatty acid profile. The results are given in Figure 1 and show that the B100s have a variety of compositions. The low-temperature properties of the B100s are listed in Table 1. Table 2 contains the low-temperature properties of the Ultra-Low Sulfur diesel (ULSD) selected for this project. Since the aromatic/saturated compound concentrations of the diesel fuel may also have an effect on low-temperature properties, the ULSD fuel used for blending was analyzed for aromatic content, using ASTM D5186. The results of this analysis are presented in Table 3.

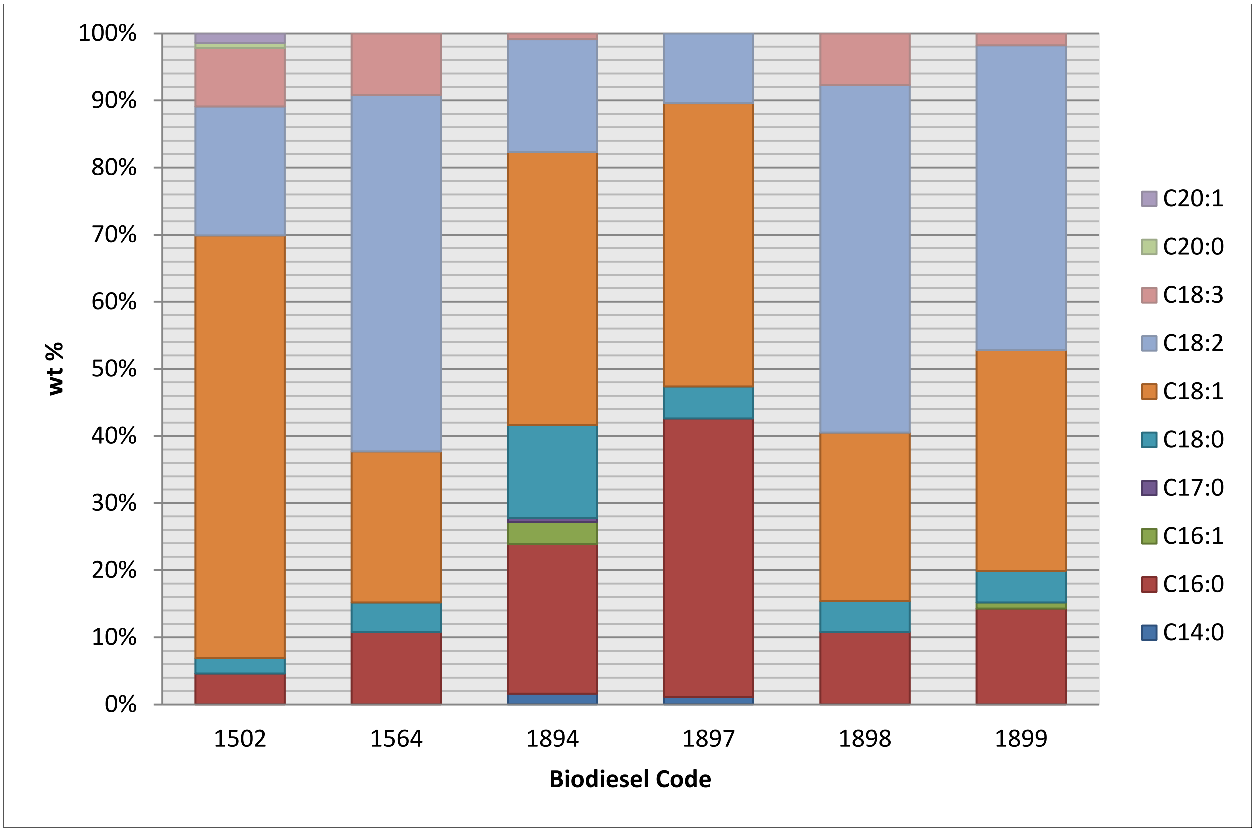

Figure 1. B100 Fatty Acid Profile

Table 1. Low Temperature properties of B100 samples

Biodiesel	Cloud Point, ˚C	Pour Point, ˚C	Cold Filter Plugging Point, ˚C	Cold Soak Filterability Test, Seconds
CL10-1502	-2.0	-12	-13	65
CL10-1564	-0.4	-3	-3.5	137
CL10-1894	11.5	12	6	122
CL10-1897	14.8	15	11	Fail
CL10-1898	0.1	-3	-6	52
CL10-1899	1.8	0	-4	87

Table 2. Low Temperature Properties of Ultra-Low Sulfur Diesel Fuel

Sample No.	Sample Description	Cloud Point, °C	Pour Point, °C	Cold Filter Plugging Point, °C
AF 7715: CL10-1751	Ultra-Low Sulfur Diesel Fuel	-15.9	-21.0	-16.0

Table 3. Aromatic Content of Ultra-Low Sulfur Diesel Fuel

Total Aromatics	**Mass%**	34.9
MonoAromatics	**Mass%**	30
PolyAromatics	**Mass%**	4.9

3.3 Low-Temperature Properties of Blends

Each of the B100s was blended at 5%, 10%, and 20% with the ULSD. These blends were then tested for CP, PP, and CFPP. The results are given in Table 4.

Table 4. Low-Temperature Properties for B5, B10, and B20

	Biodiesel blends of CL10-1502 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-14.7	-13.6	-11.9
Pour Point, °C	-24.0	-24.0	-21.0
Cold Filter Plugging Point, °C	-18.0	-18.0	-21.0
	Biodiesel blends of CL10-1564 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-14.7	-13.8	-12.6
Pour Point, °C	-24.0	-24.0	-18.0
Cold Filter Plugging Point, °C	-16.5	-16.5	-15.0
	Biodiesel blends of CL10-1894 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-14.3	-12.5	-9.3
Pour Point, °C	-24.0	-21.0	-12.0
Cold Filter Plugging Point, °C	-16.0	-15.0	-13.0

Table 4. Low-Temperature Properties for B5, B10, and B20 (Continued)

	Biodiesel blends of CL10-1897 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-13.6	-12.7	-6.8
Pour Point, °C	-21.0	-18.0	-12.0
Cold Filter Plugging Point, °C	-25.0	-21.0	-10.0
	Biodiesel blends of CL10-1898 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-14.1	-13.4	-11.9
Pour Point, °C	-24.0	-21.0	-18.0
Cold Filter Plugging Point, °C	-19.0	-20.0	-21.0
	Biodiesel blends of CL10-1899 / ULSD		
Properties	**B5**	**B10**	**B20**
Cloud Point, °C	-14.6	-13.4	-11.3
Pour Point, °C	-21.0	-24.0	-21.0
Cold Filter Plugging Point, °C	-21.0	-28.0	-21.0

3.4 Additives Used

Four additives were selected for evaluation in this work, they are identified below:

- Additive 1: R488
- Additive 2: Viscoplex 10/34
- Additive 3: Viscoplex 10/6830
- Additive 4: Bio 9928

These additives were selected based on the results of another study, conducted by DLA Energy, to evaluate low-temperature additives.[i]

3.5 Additized Blends

Blends of the six (6) B100 samples were additized with the additives selected for this effort, and subsequently analyzed for their low-temperature properties. The additized blends were made at room temperature (representing optimum blending conditions). The additives were added to the unadditized samples, and allowed to mix for thirty minutes before analysis. The changes in the cloud points, pour points, and cold filter plugging points of the B20 and B10 samples after additization are included in Figure 2 through Figure 10.

As a general rule, the results show that when the additives are blended under optimum conditions, Additives 2 and 3 give the most improvement in CP, while Additives 1 and 4 have the greatest effect on PP and CFPP.

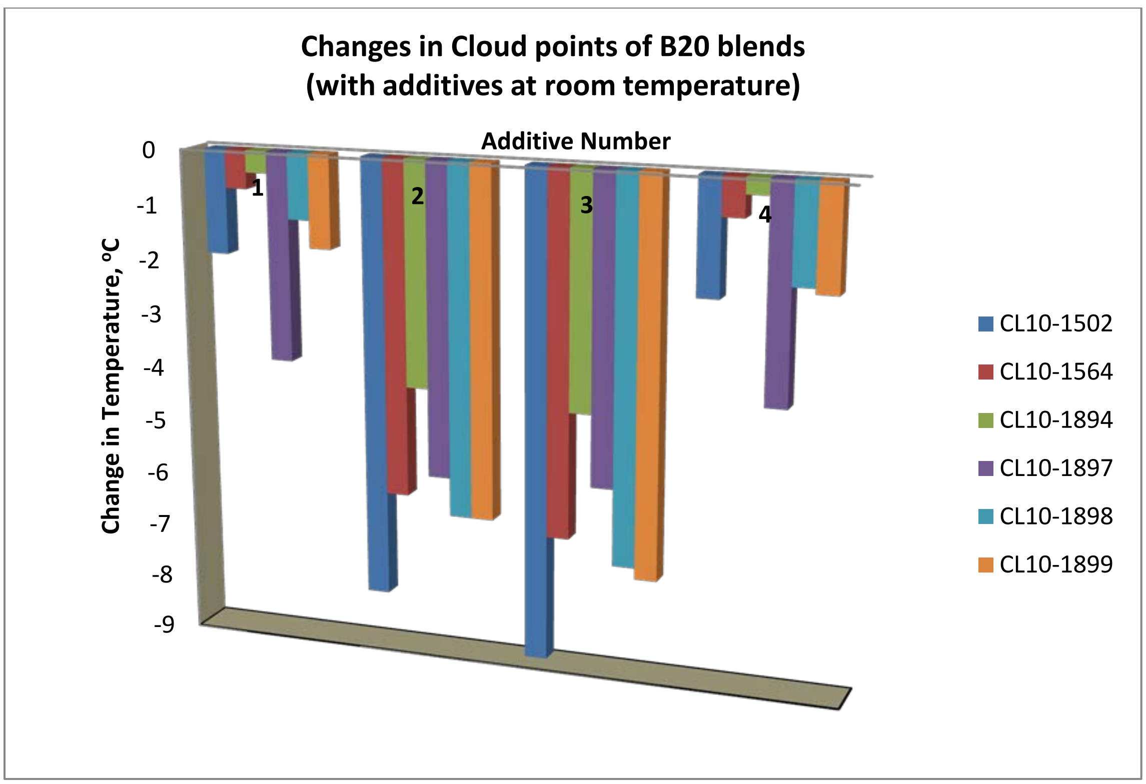

Figure 2. Differences in B20 Cloud Points with Additives

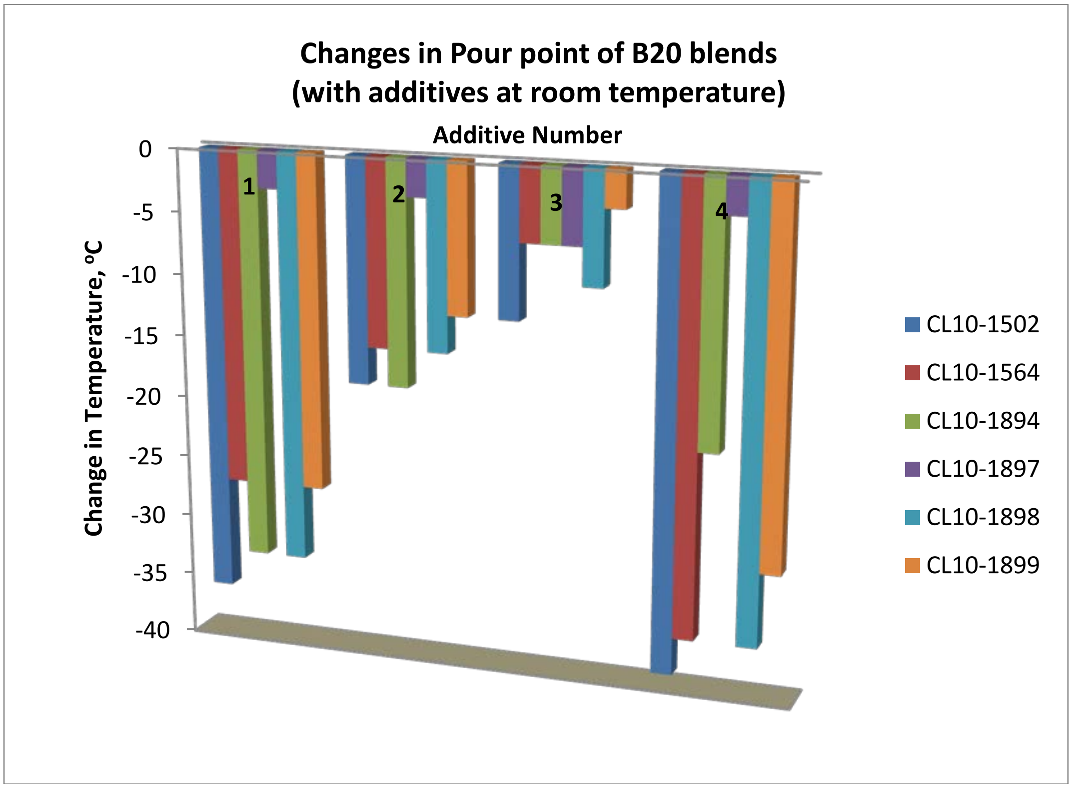

Figure 3. Differences in B20 Pour Points with Additives

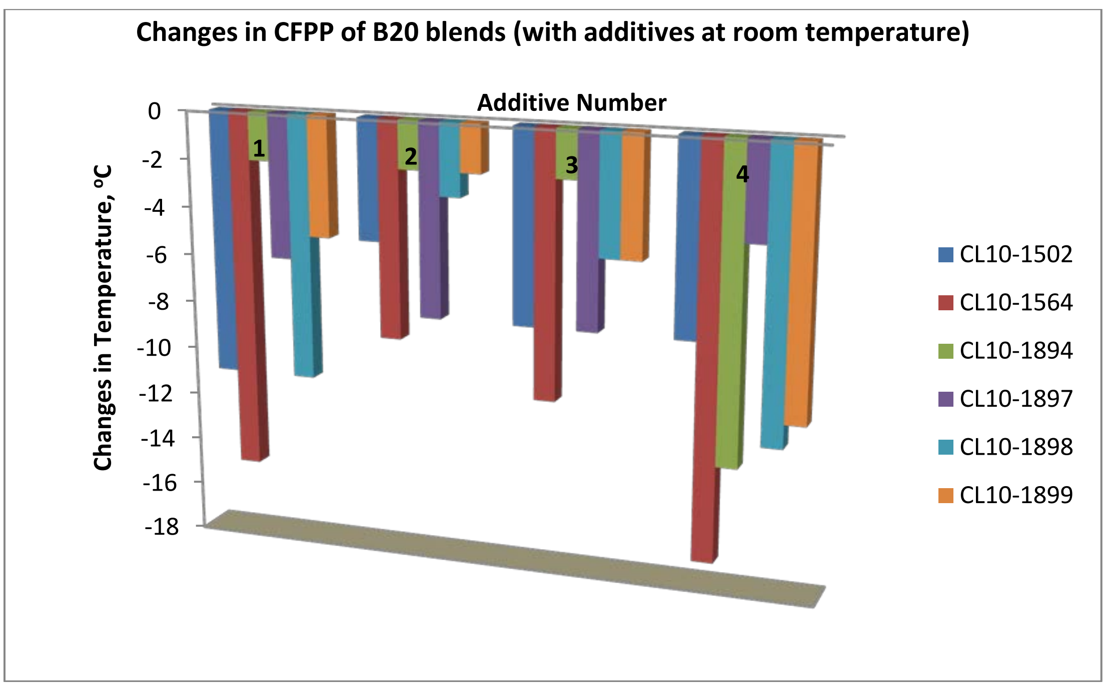

Figure 4. Differences in B20 Cold Filter Plugging Points with Additives

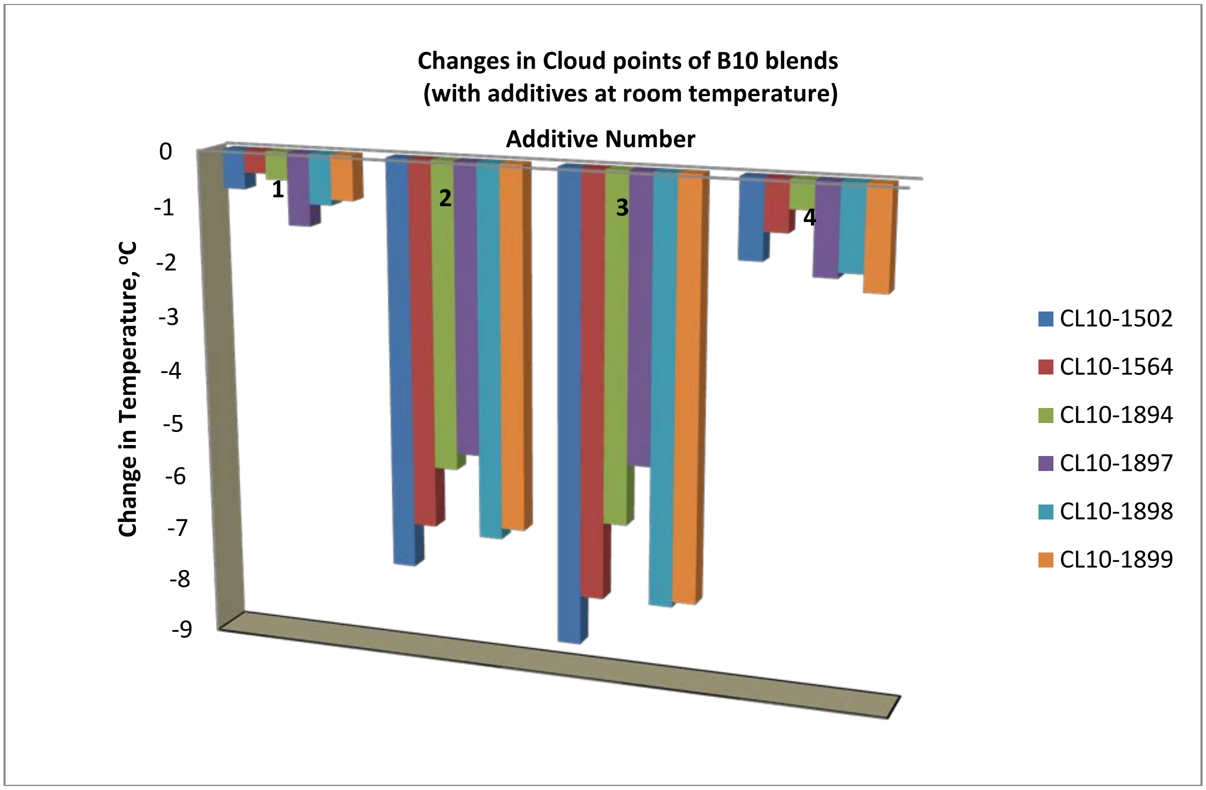

Figure 5. Differences in B10 Cloud Points with Additives

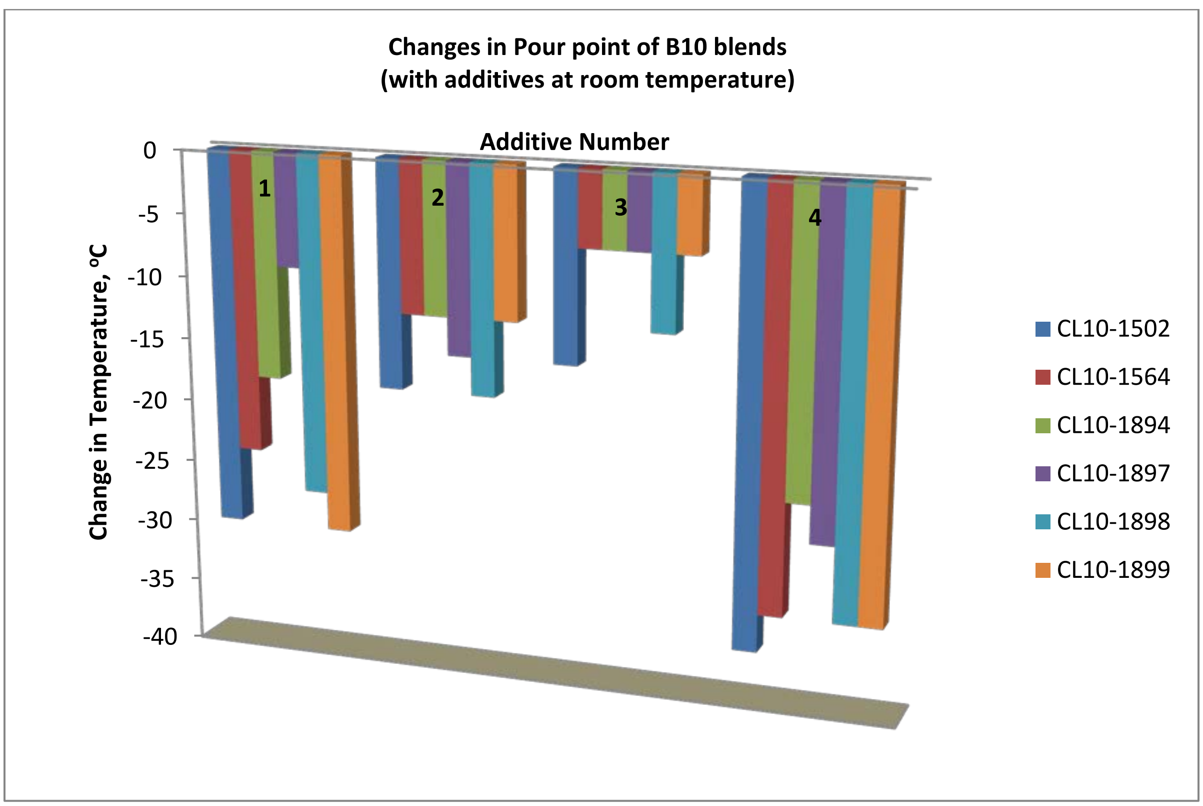

Figure 6. Differences in B10 Pour Points with Additives

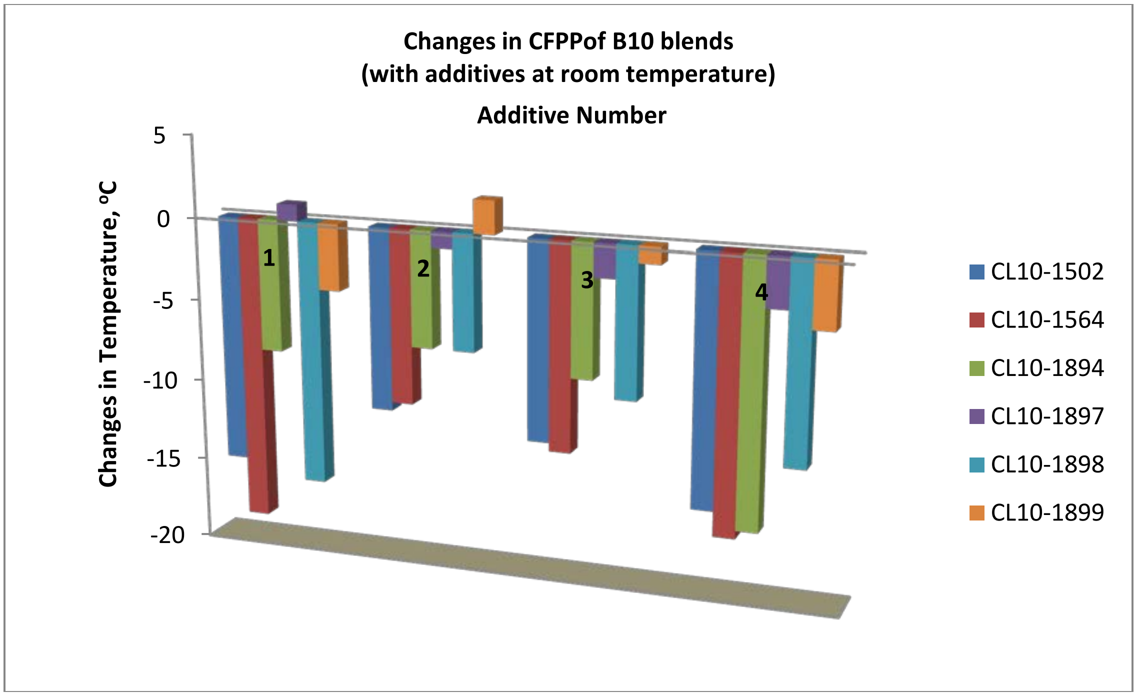

Figure 7. Differences in B10 Cold Filter Plugging Points with Additives

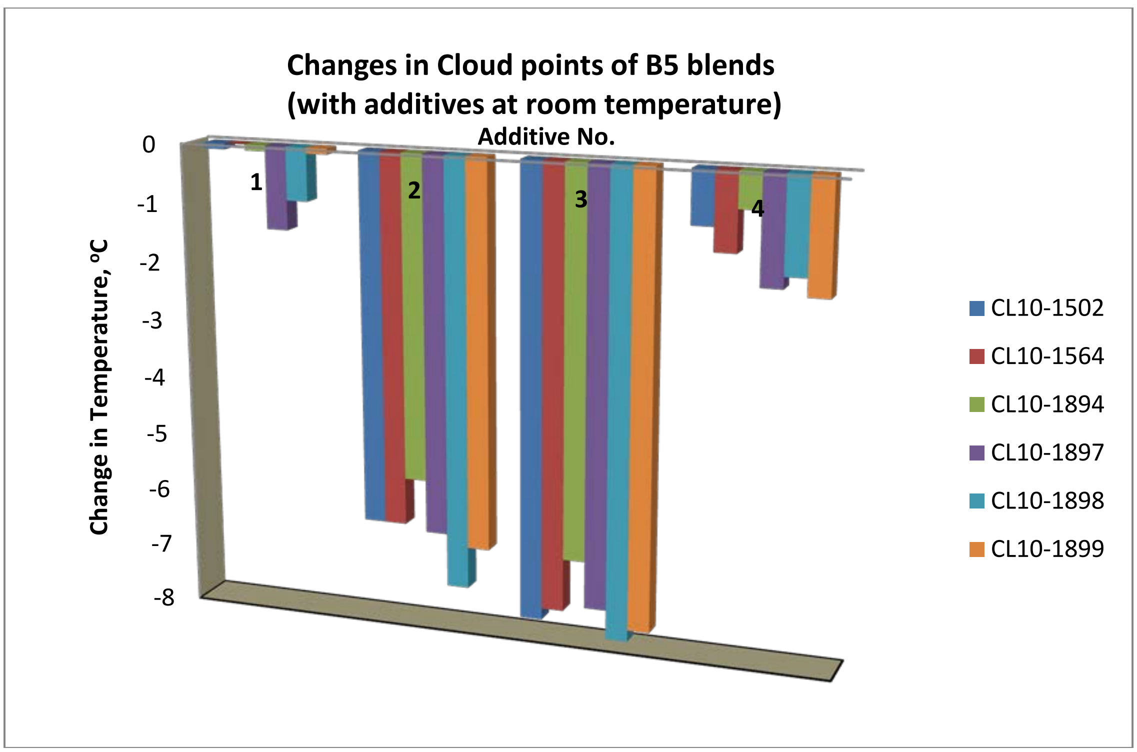

Figure 8. Differences in B5 Cloud Points with Additives

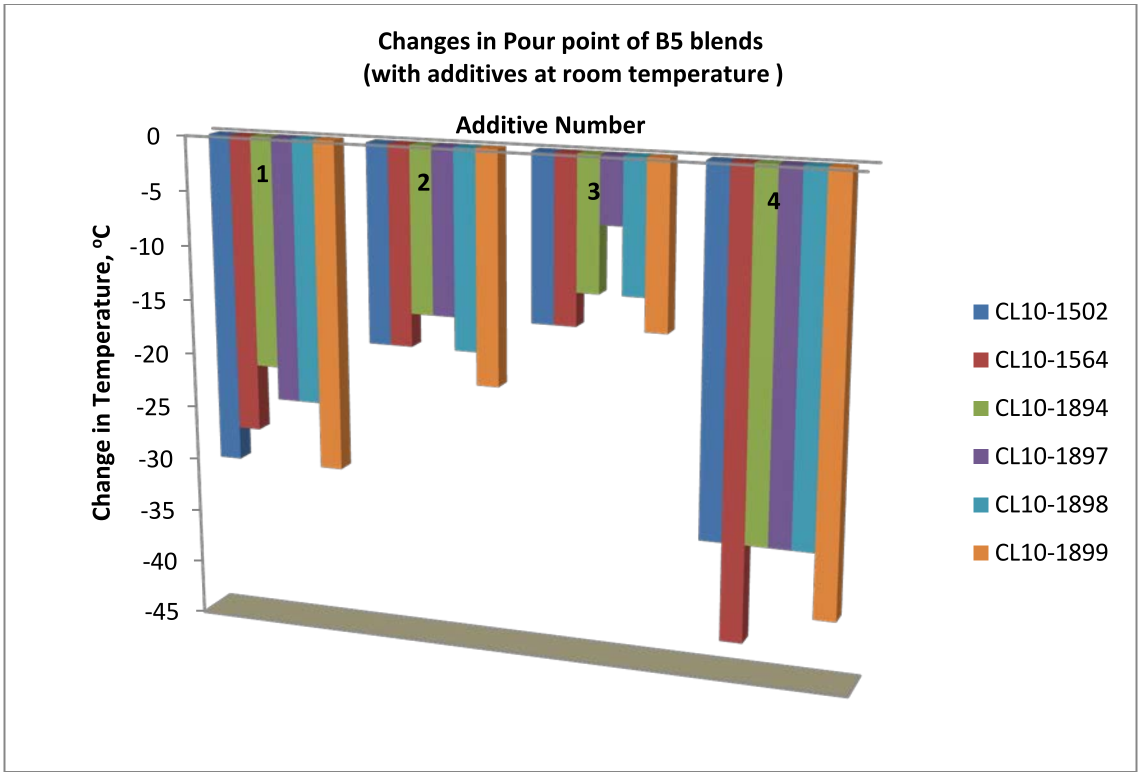

Figure 9. Differences in B5 Pour Points with Additives

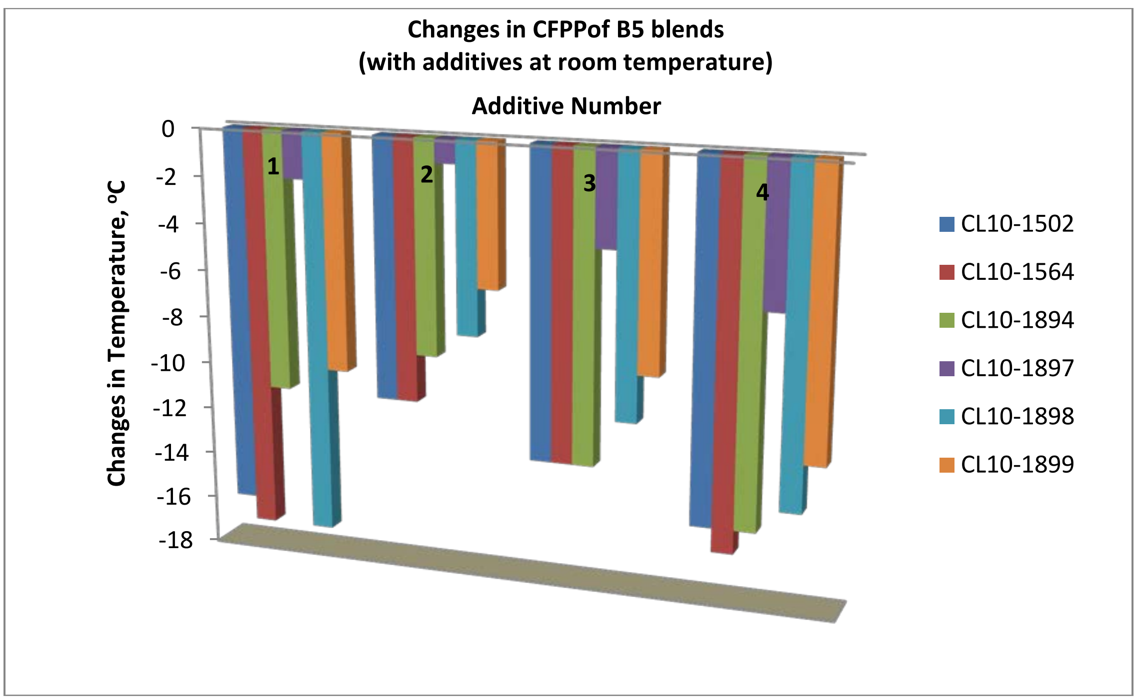

Figure 10. Differences in B5 Cold Filter Plugging Points with Additives

3.6 Additization at Low Temperatures

It is expected that biodiesel blend users may often be required to additize blends under low-temperature (i.e. less than optimum) conditions. Following consultations with Army representatives, it was decided to investigate the possibility of assembling a test apparatus to evaluate techniques for adding low-temperature operability additives to cold fuel. Figure 11 is a photograph of the apparatus that was assembled. Limited proof-of-concept testing was conducted with the apparatus. A description of the device and the testing conducted with it are given below.

Description of Apparatus: A 500 ml beaker of fuel, filled with approximately 300 ml of fuel, was placed inside an empty larger beaker. The larger beaker was to act as an air barrier. The two beakers were then placed inside a large glass jar. Dry ice was placed inside between the glass jar and the larger air barrier beaker. Dry ice acted as a sufficient cooling agent for this experiment. The dry ice was placed throughout the jar and up to the fill line of the fuel in an attempt to ensure temperatures were uniform throughout the sample. A thermocouple and the filter, which was attached to a steel rod, a pipette, and vacuum source, were then placed inside the fuel sample. The thermocouple and filter apparatus were attached securely to the lid of the glass jar for stability.

Experiment: We obtained a fuel sample (B20) with a known CFPP of -21°C. This sample contained no additive and was tested in the device. We cooled the sample to -21°C, applied a vacuum to the pipette and filter apparatus, and the sample plugged accordingly. We then let the sample warm to room temperature then cooled it back down to the cloud point of the sample of -12°C. An additive, mixed with 1% of diesel fuel, was then injected into the fuel sample, using a pipette, through a prepared opening in the lid of the glass jar. There was no mixing of the additive after injection. The sample (with the additive) was then cooled to its original CFPP of -21°C to test and see if the additive made improvement of the CFPP. A vacuum was applied but no fuel aspirated through the pipette. This sample was warmed to room temperature, approximately 22°C. The sample was then stirred vigorously to ensure the additive was mixed into the fuel. (The fuel temperature was measured after mixing.) The sample was then placed back in the device and cooled to its original CFPP of -21°C. A vacuum was applied for aspiration of the sample at this point to see if the additive (with mixing) made an improvement of the CFPP. There was slight improvement, as the sample plugged at -25°C. This sample (with the additive mixed under optimum conditions) had been previously tested and resulted in a CFPP of -32°C.

The above described experiment was repeated with several other additive / biodiesel blend combinations. The results were inconsistent, owing to the fact that we could not accurately control the conditions in the fuel beaker. However, all of the tests showed that attempts to mix additives into cold fuel usually result in no improvement in the low-temperature properties of the biodiesel blend.

Figure 11. Test Assembly for Low Temperature Fuel Additization

CFPP of additized blends with adequate and minimal mixing were evaluated using only the automated instrument. This was also combined with studying the effect of temperature in additive performances. The objective of the study was to develop a procedure to test additives at temperatures lower than the room temperature with simulation of conditions close to the real world.

With this objective, three different B20 biodiesel blends were tested using a technique used to simulate conditions close to the field. Additive #4 was used for all the tests, and it was dissolved in 1% of Ultra-Low Sulfur Diesel (ULSD) fuel prior to addition to the biodiesel samples. The automatic CFPP instrument is a multi-stage (two stages) instrument where each stage operation can be achieved separately. In the first stage, unadditized biodiesel blends were tested at room temperature to determine their CFPP's. Subsequently, the additives were added to the cold biodiesel blend and were retested. Following evaluation of the CFPP, the samples were allowed to warm to room temperature, and retested. The results of this evaluation are included in Table 5. Samples in stage #2 were cooled to temperatures higher than the CFPP and subsequently the additives were added to the blend. During this testing, samples were adequately mixed by the action of the testing process.

Table 5. Effect of Temperature on Additive (#4) Performances, with Adequate Mixing (B20)

Biodiesel Description	CFPP, °C (without additive)	CFPP, °C Stage #1	CFPP, °C (after sample allowed to warm to room temperature)	CFPP, °C (with additive addition at temperatures three degrees higher than CFPP with adequate mixing)- Stage #2
B20 : CL10-1894	-14	-17	-19	-17
B20 : CL10-1898	-20	-21	-33	-35
B20 : CL10-1899	-20	-20	-33	---
B20 : CL10-1897	-11	-13	-14	-15
B20 : CL10-1502	-26	-24	-21	-25
B20 : CL10-1564	-18	-16	-34	-32

Cold Filter Plugging Point (CFPP) of additized (#4) blends, B5, and B10 with adequate and minimal mixing were evaluated using an automated instrument. The results of the analysis of six (6) biodiesel samples are included in Table 6 and Table 7 respectively.

Table 6. Effect of Temperature on Additive Performances, with Adequate Mixing (B5 Blends)

Biodiesel Description	CFPP, °C (without additive) Stage #1	CFPP, °C Stage #1	CFPP, °C (after sample warmed to room temperature) Stage#1	CFPP, °C (with additive at temperatures 3 degrees higher than CFPP with adequate mixing) Stage #2
CL10-1502	-20	-18	-35	-34
CL10-1564	-17	-34	-34	-33
CL10-1894	-16	-32	-33	-33
CL10-1897	-25	-23	-33	-24
CL10-1898	-20	-18	-36	-35
CL10-1899	-20	-34	-36	-34

Table 7. Effects of Temperature on Additive (#4) Performance, with Minimal Mixing (B5 Blends)

Biodiesel Description	CFPP, °C (without additive)	CFPP, °C (with additive addition at temperatures close to CFPP)
CL10-1502	-20	-34
CL10-1564	-17	-33
CL10-1894	-16	-28
CL10-1897	-25	-25
CL10-1898	-20	-22
CL10-1899	-20	-22

The aforementioned experiments were repeated with the B10 blends; and results of these analyses are included in Table 8 and Table 9.

Table 8. Effect of Temperature on Additive (#4) Performances, with Adequate Mixing (B10 Blends)

Biodiesel Description	CFPP, ^oC (without additive) Stage #1	CFPP, ^oC Stage #1	CFPP, ^oC (after sample warmed to room temperature) Stage#1	CFPP, ^oC (with additive at temperatures 3 degrees higher than CFPP with adequate mixing) Stage #2
CL10-1502	-19	-31	-33	-33
CL10-1756	-18	-29	-31	-16
CL10-1894	-15	-27	-28	-31
CL10-1897	-22	-26	-23	-20
CL10-1898	-19	-31	-31	-35
CL10-1899	-21	-32	-33	-35

Table 9. Effects of Temperature on Additive (#4) Performance, with Minimal Mixing (B10 Blends)

Biodiesel Description	CFPP, ^oC (without additive)	CFPP, ^oC (with additive addition at temperatures close to CFPP)
CL10-1502	-19	-22
CL10-1756	-18	-21
CL10-1894	-15	-29
CL10-1897	-22	-22
CL10-1898	-19	-33
CL10-1899	-21	-22

The results of both analyses, B5 and B10, indicated that the performance of additive #4 was less influenced by concentration of biodiesel in the blends than by the temperature and type of additive mixing.

Additive No. 2 was evaluated for its improvement in Cold Filter Plugging Point (CFPP) of biodiesel blends, with adequate and minimal mixing, using an automated instrument. The results of the analysis of the six (6) biodiesel blends in varying percentages of biodiesel are included in Table 10 through Table 15, respectively.

The test results of the B5 blends using additive No. 2 are shown below in Table 10 and Table 11.

Table 10. Effect of Temperature on Additive No. 2 Performances, with Adequate Mixing (B5 Blends)

Biodiesel Description	CFPP, ^oC (without additive) Stage #1 Test CFPP	CFPP, ^oC Stage #1 Inject additive after CFPP Start new test 2 degrees before CFPP	CFPP, ^oC (after sample warmed to room temperature) Stage#1	CFPP, ^oC (with additive at temperatures 2 degrees higher than CFPP with adequate mixing) Stage #2 Inject additive before CFPP
CL10-1502	-19	-27	-29	-25
CL10-1564	-19	-17*	-28	-26
CL10-1894	-16	-25	-26	-26
CL10-1897	-21	-19*	-25	-21
CL10-1898	-19	-17*	-28	-27
CL10-1899	-20	-21	-27	-24

*sample plugged on initial aspiration of test

Table 11. Effects of Temperature on Additive No. 2 Performance, with Minimal Mixing (B5 Blends)

Biodiesel Description	CFPP, ^oC (without additive)	CFPP, ^oC (with additive addition at temperatures close to CFPP) Inject additive at CFPP
CL10-1502	-19	-20
CL10-1564	-19	-19
CL10-1894	-16	-22
CL10-1897	-21	-21
CL10-1898	-19	-19
CL10-1899	-20	-21

The test results of the B10 blends using additive No. 2 are shown below in Table 12 and Table 13.

Table 12. Effect of Temperature on Additive No. 2 Performances, with Adequate Mixing (B10 Blends)

Biodiesel Description	CFPP, °C (without additive) Stage #1 Test CFPP	CFPP, °C Stage #1 Inject additive after CFPP Start new test 2 degrees before CFPP	CFPP, °C (after sample warmed to room temperature) Stage#1	CFPP, °C (with additive at temperatures 2 degrees higher than CFPP with adequate mixing) Stage #2 Inject additive before CFPP
CL10-1502	-19	* -17	-29	-21
CL10-1564	-18	-23	-27	-25
CL10-1894	-15	-22	-23	-22
CL10-1897	-20	-18	-23	-18
CL10-1898	-19	* -17	-27	-26
CL10-1899	-21	-19	-27	-19

*sample plugged on initial aspiration of test

Table 13. Effects of Temperature on Additive No. 2 Performance, with Minimal Mixing (B10 Blends)

Biodiesel Description	CFPP, °C (without additive)	CFPP, °C (with additive addition at temperatures close to CFPP) Inject additive at CFPP
CL10-1502	-19	-19
CL10-1564	-18	-18
CL10-1894	-15	-15
CL10-1897	-20	-20
CL10-1898	-19	-19
CL10-1899	-21	-21

The test results of the B20 blends using additive No. 2 are shown below in Table 14 and Table 15.

Table 14. Effect of Temperature on Additive No. 2 Performances, with Adequate Mixing (B20 Blends)

Biodiesel Description	CFPP, °C (without additive) Stage #1 Test CFPP	CFPP, °C Stage #1 Inject additive after CFPP Start new test 2 degrees before CFPP	CFPP, °C (after sample warmed to room temperature) Stage#1	CFPP, °C (with additive at temperatures 2 degrees higher than CFPP with adequate mixing) Stage #2 Inject additive before CFPP
CL10-1502	-28	* -26	-28	-26
CL10-1564	-18	* -16	-25	-16
CL10-1894	-15	* -13	-16	-13
CL10-1897	-11	* -9	-18	-17
CL10-1898	-19	* -17	-24	-18
CL10-1899	-21	* -19	-23	-19

*sample plugged on initial aspiration of test

Table 15. Effects of Temperature on Additive No. 2 Performance, with Minimal Mixing (B20 Blends)

Biodiesel Description	CFPP, °C (without additive)	CFPP, °C (with additive addition at temperatures close to CFPP) Inject additive at CFPP
CL10-1502	-28	-28
CL10-1564	-18	-18
CL10-1894	-15	-15
CL10-1897	-11	-11
CL10-1898	-19	-19
CL10-1899	-21	-21

Observations recorded while testing performance of additive No. 2 were similar to those recorded while testing additive No. 4. The effectiveness of additives in improving the cold temperature properties of cold biodiesel fuel blends is highly dependent on the degree of mixing provided.

4.0 CONCLUSIONS

The following conclusions are based on the results presented herein:

- B100s and biodiesel blends with varying fatty acid profiles were used to evaluate the selected additives.
- Additives 1 and 4 performed best to improve PP and CFPP.
- Additives 2 and 3 performed best to improve CP.
- In general, the performance of the selected additives was less effected by the fatty acid profile than by the test method used to evaluate the additive.
- In general, the concentration of biodiesel in the blends did not affect the efficacy of a given additive; but did influence the magnitude of the effect.
- The results confirmed, as expected, that the additives performed best when mixed into the blend under optimum conditions.
- Modified CFPP tests suggest that the efficacy of additives blended into cold fuel can generally be improved by minimal agitation and/or warming of the fuel during additization.
- There is no useful parameter to utilize as a means to determine whether to use additive or not. Most fuel will show some improvement in low-temperature operability after treatment with the most effective additives evaluated above. The only way to confirm actual performance improvements is to test the fuel with and without an additive.

5.0 REFERENCES

i Moorthy, K. and Westbrook, S., “Evaluations of Low Temperature Operability Additives for Biodiesel Blends,” DLA Energy, January 2011.